

**Steel for mechanical construction, method of hot-shaping of a part from this steel, and part thus obtained.**

Background of the invention

The invention relates to the metallurgy of iron and steel, and more precisely to the manufacture of parts made from steel which can in particular be used in mechanical construction and shaped by the process known as "thixoforging".

Thixoforging belongs to the category of processes for shaping metals in the semi-solid state.

This process consists of producing a substantial deformation on a billet heated between the solidus and the liquidus.

The steels used for this process are those which are conventionally used for hot-forging, and which are if necessary previously subjected to a metallurgical operation consisting of globulising the primary structure which is conventionally dendritic. In fact, this dendritic primary structure is not adapted to the thixoforging operations. In the course of heating up to temperatures between the solidus and the liquidus, the micro-segregation existing between the dendrites and the inter-dendritic spaces will bring about the fusion of the steel preferentially in these inter-dendritic spaces. During the operation of shaping this intergrowth of liquid and solid, the liquid phase will be ejected in a first stage at the start of the application of force. Therefore it is necessary to deform the solid phase and a residue of liquid for the most separated from the solid phase, which will result in an increase in the forces. For a deformation operation under these conditions the result obtained is poor: substantial segregation, internal defects.

On the other hand, when the thixoforging is carried out on a steel of globular structure brought to the semi-solid state by heating at a temperature between the liquidus and the solidus, the globular solid particles are distributed uniformly in the liquid phase. By optimising the choice of the solid/liquid proportions, it is possible to obtain a material having

a raised rate of deformation under the effect of a considerable shear stress. It therefore has a very high deformability.

However, it is possible in certain cases to obtain the desired globular structure in the course of heating prior to the thixoforging, without having to carry out an operation of globulisation of the separated primary structure. This is the case in particular when operating on billets produced from rolled bars derived from continuous casting blooms or ingots. The multiple reheating and substantial deformations undergone by the steel have then led to a very imbricate and diffuse structure where a primary structure is practically impossible to show. It makes it possible to obtain a globular structure of the solid phase during the heating prior to thixoforging.

Thus the thixoforging makes it possible, by comparison with conventional hot-forging processes, to produce in one single deformation operation parts of complex geometry which may have thin walls (1mm or less) with very low shaping forces. In fact, under the action of external forces steels suitable for a thixoforging operation behave like viscous fluids.

For steels for mechanical construction, in which the carbon content can vary from 0.2% to 1.1%, the heating temperature necessary for the deformation by the thixoforging process is for example  $1430^{\circ}\text{C} + 50^{\circ}\text{C} = 1480^{\circ}\text{C}$  (measured solidus temperature +  $50^{\circ}\text{C}$  to obtain the good ratio of liquid phase to solid phase necessary for the deformation) and  $1315^{\circ}\text{C} + 50^{\circ}\text{C} = 1365^{\circ}\text{C}$  for a grade 100Cr6.

The heating temperature and the quantity of liquid phase formed are important parameters of the thixoforging process. The ease of obtaining the "good" temperature and the range of dispersion about this temperature so as to limit the variations of the quantity of liquid phase depend upon the solidification range. The greater this range is the easier it is to regulate the heating parameters.

For example, this solidification range is 110°C for a grade C38 and 172°C for the grade 100Cr6. Therefore it is much easier to work with this latter grade which has a low solidus temperature: 1315°C and a large solidification range: 172°C.

The very high shaping temperatures, the substantial rates of deformation which are used in the thixoforging process, lead to thermal stress on the deformation tools under conditions which are frequently extreme. This leads to the use for these tools of alloys with very high mechanical characteristics when hot or of ceramic materials. The difficulties of producing certain geometries or tools (inserts) of substantial volumes and the costs of producing them can slow down the development of the thixoforging process.

The object of the invention is to propose new grades of steel which are better adapted to thixoforging than those which are used conventionally in that they would make it possible to reduce the stresses on the deformation tools. Moreover, these new grades should not degrade the mechanical properties of the parts obtained.

#### Brief summary of the invention

To this end, the invention relates to a steel for mechanical construction, wherein its composition in percentages by weight is:

- $0.35\% \leq C \leq 1.2\%$
- $0.10\% \leq Mn \leq 2.0\%$
- $0.10\% \leq Si \leq 3.0\%$
- $traces \leq Cr \leq 4.5\%$
- $traces \leq Mo \leq 2.0\%$
- $traces \leq Ni \leq 4.5\%$
- $traces \leq V \leq 0.5\%$
- $traces \leq Cu \leq 3.5\%$  with  $Cu \leq Ni\% + 0.6 Si\%$  if  $Cu \geq 0.5\%$
- $traces \leq P \leq 0.200\%$ ,  $traces \leq Bi \leq 0.200\%$ ,  $traces \leq Sn \leq 0.150\%$ ,  $traces \leq As \leq 0.100\%$ ,  $traces \leq Sb \leq 0.150\%$ , with  $0.050\% \leq P\% + Bi\% + Sn\% + As\% + Sb\% \leq 0.200\%$ ,
- $traces \leq Al \leq 0.060\%$
- $traces \leq Ca \leq 0.050\%$

- traces  $\leq B \leq 0.01\%$
- traces  $\leq S \leq 0.0200\%$
- traces  $\leq Te \leq 0.020\%$
- traces  $\leq Se \leq 0.040\%$
- traces  $\leq Pb \leq 0.070\%$
- traces  $\leq Nb \leq 0.050\%$
- traces  $\leq Ti \leq 0.050\%$

the remainder being iron and impurities resulting from the manufacture.

According to a variant of the invention, its Si content is between 0.10% and 1.0%.

The ratio Mn%/Si% is preferably greater than or equal to 0.4.

The invention also relates to a method of hot-shaping a steel part, wherein:

- a billet of steel of the preceding composition is obtained;
- a heat treatment is if need be applied to it, which gives it a globular primary structure;
- it is heated to an intermediate temperature between its solidus temperature and its liquidus temperature under conditions such that the solid fraction has a globular structure;
- thixoforging of the said billet is carried out so as to obtain the said part;
- and cooling of the said part is carried out.

The said thixoforging takes place preferably in a zone of temperatures where the liquid material fraction present in the billet is between 10 and 40%.

The said cooling is preferably carried out in still air.

The said cooling may be effected at a speed lower than that which would obtain natural cooling in air.

The invention also relates to a part made from thixoforged steel, wherein it has been manufactured by the preceding method.

As will be understood, the invention consists essentially of adding to a steel for mechanical construction having the usual composition one or several elements chosen from amongst phosphorus, bismuth, tin, arsenic and antimony, and also silicon, in defined proportions. These analytical modifications render the steel particularly well adapted to shaping of the part made from it by thixoforging.

#### Brief description of the several views of the drawings

The invention will be better understood on reading the following description which is given with reference to the accompanying Figure 1 which shows the proportion of liquid phase in the steel as a function of the temperature for a reference steel and for a steel according to the invention, and with reference to Figure 2 which shows the same values for another pair of reference steel and steel according to the invention.

#### Detailed description of the invention

In order to reduce the stresses on the tools during the thixoforging and to make this easier, the person skilled in the art has a first solution which consists, as has been said, of lowering the working temperatures by the addition of carbon. This solution makes it possible to lower the liquidus and solidus temperatures. However, it has the drawback that it has a substantial influence on the mechanical properties of the steel.

The inventors imagined that a beneficial effect on the stresses could be obtained by the addition of elements having a strong tendency to segregation at the grain boundaries. This strong segregation is not usually sought. In fact, the fusion of such segregated zones at a temperature lower than the solidus, generally called the burning temperature, is prejudicial to the conventional hot-shaping operations: rolling and forging.

For a given forging or rolling temperature, lower than the solidus temperature for the matrix of the metal to be deformed, the presence of liquid zones due to elements which segregate at low fusion points, even with very small volumes (a few %) at the solid grain boundaries will lead to the disaggregation of the shaped material; this is the solid part which controls the

deformation mechanisms for these shaping methods, and the forces necessary for shaping lead to (partial or total) ruptures of material which are prejudicial to the production of the product and to its properties. In the case where the liquid phase is greater than 10%, which is the case in thixoforging, the material is two-phase, which results in very different behaviour during the deformation: the solid particles are included in liquid and if there are contacts (called bridges) between the solid particles the very weak forces necessary to rupture them do not cause ruining of the material.

In the case of thixoforging where the burning temperature is greatly exceeded, the fusion of the segregated zones creates liquid pockets which favour and accelerate the formation of liquid phase within the steel. Therefore there is an interest in promoting this.

Thus it is possible to obtain the quantity of liquid phase necessary for the thixoforging to proceed well at a temperature lower than that usually necessary when the process does not go on to the addition of at least one of the elements phosphorus, bismuth, tin, arsenic or antimony when the sum of the contents of these elements is at least 0.050%.

The sum of the elements phosphorus, bismuth, tin, arsenic and antimony must not exceed 0.200% so as to avoid the problems mentioned above during hot-rolling or forging, enabling the billet to be obtained which is intended to undergo thixoforging.

Naturally, in the case of addition of arsenic during the production of the liquid metal, all the necessary precautions must be taken so that the toxic vapours released are collected in such a manner that they do not poison the staff at the steelworks. In fact the presence of arsenic most frequently results from the addition of copper or tin which arsenic generally accompanies by way of an impurity. As arsenic is an element which is very highly segregating, it is necessary to take it into account to be sure that in combination with the other segregating elements it does not lead to effects which are prejudicial to the hot transformation which has been cited.

The carbon content of the steels according to the invention can vary between 0.35% and 1.2%. Under these conditions it is possible to obtain metallurgical structures, mechanical properties and wear properties which are desirable for thixoforged steel parts which can be used in mechanical construction. The carbon content must be chosen as a function of the use envisaged.

The silicon content of the steels according to the invention can vary typically between 0.10 and 1.0%, but may go up to 3.0% if a particularly accentuated effect is required from the addition of segregating elements and if the cost of the massive addition of silicon does not appear prohibitive to the manufacturer. Like carbon, silicon makes it possible to lower the solidus and liquidus temperatures and to widen the solidification range. It also has a synergetic effect on the segregation of the other elements. Furthermore it makes it possible to improve the fluidity of the metal.

The manganese content can be between 0.10 and 2.0%. It must be adjusted as a function of the mechanical properties required, in conjunction with the carbon and silicon contents. It has relatively little influence on the liquidus and solidus temperatures. But if the fluidity is raised because of a high silicon content (for example 1% or more), a manganese content which is too low gives the metal insufficient mechanical properties in the course of cooling during continuous casting, and hence a risk of the appearance of cracks. Such cracks can also appear for the same reasons during cooling following thixoforging, all the more so as the great variations in thickness of the part lead to significant disparities over the local cooling speeds. Thus stresses are created which are likely to favour the appearance of cracks if the mechanical properties of the steel are insufficient. For these reasons it is preferable for the ratio Mn%/Si% to be greater than or equal to 0.4.

The chromium content may be between traces and 4.5%.

The molybdenum content may be between traces and 2.0%.

The nickel content may be between traces and 4.5%.

The adjustment of the chromium, molybdenum and nickel contents makes it possible to ensure the mechanical properties of the parts produced: resistance to rupture, yield strength limit and resilience.

The vanadium content is between traces and 0.5%.

For certain applications where the resilience is not important, this element makes it possible to obtain steels with very high mechanical characteristics which can be substituted for steels rich in chromium and/or molybdenum and/or nickel, which are more expensive.

The copper content may be between traces and 3.5%. This element makes it possible to increase the mechanical characteristics, to improve the corrosion resistance and to lower the solidus temperature. It should be noted that if copper is present in high quantities (0.5% and more) it is necessary for nickel and/or silicon to be present in sufficient quantities to avoid problems on hot-rolling or forging. It is considered that if  $\text{Cu}\% \geq 0.5\%$  it is necessary for  $\text{Cu} \leq \text{Ni}\% + 0.6 \text{ Si}\%$ .

With regard to the segregating elements, the presence of which is typical of the invention, the sum of the phosphorus, bismuth, tin, arsenic and antimony contents must be at least 0.050% and must not exceed 0.200%. These elements can be present alone or in combination. If they are alone (that is to say that the other elements in the list are only present as traces), then there must be at least 0.050% of phosphorus, or 0.050% of bismuth, or 0.050% of tin, or 0.050% of arsenic or 0.050% of antimony.

The contents of aluminium and calcium, deoxidising elements, are between traces and respectively 0.060% for aluminium and 0.0050% for calcium.

The content of boron, a hardening element, is between traces and 0.010%.



The sulphur content is between traces and 0.200%. A high content favours the machinability of the metal, particularly if it has added to it elements such as tellurium (up to 0.020%), selenium (up to 0.040%) and lead (up to 0.070%). These elements for machinability have only a little influence on the solidus and liquidus temperatures. When sulphur is added in significant quantities, it is good to have a ratio Mn%/S% of at least 4 so that the hot-rolling is carried out without the formation of defects.

Niobium and titanium, when they are added, make it possible to control the grain size. Their maximum admissible contents are 0.050%.

Examples of compositions of steel according to the invention and of reference steels which can be used successfully to produce thixoforged parts are given in Table 1, together with the mechanical characteristics Re (yield strength) and Rm (tensile strength) obtained on thixoforged parts after cooling in still air. The percentages are by weight and expressed in 10<sup>-3</sup>%, Re and Rm are expressed in MPa.

Table 1: Compositions of samples of steels according to the invention and reference steels (in 10<sup>-3</sup>%) and their mechanical characteristics (in MPa)

No.	C	Mn	Si	Cr	Mo	Ni	V	Cu	S	Al	P	Re	Rm
1	502	1391	200	164	<5	152	<5	194	315	<0.3	15	423	773
2	493	1451	990	156	<5	152	2	201	302	1	26	510	852
3	505	1420	256	166	<5	159	<5	196	287	3	55	455	856
4	526	1478	255	156	<5	150	<5	200	315	2	97	482	866
5	508	1425	220	164	<5	155	121	203	306	7	58	583	877
6	500	1209	279	153	<5	155	7	204	83	21	99	484	871
7	508	1178	202	108	<5	158	6	204	70	25	187	528	885
8	496	1454	945	156	<5	158	<5	202	291	<0.3	55	498	877

In these examples the steels according to the invention (Nos. 3 to 8) have undergone an addition of phosphorus bringing the content of this element to between 0.050 and 0.200%. Relative to the two reference steels with a low phosphorus content (0.015 and 0.026%), no deterioration in the mechanical properties is noted.

Table 2 shows the composition of a reference steel and of a steel according to the invention which is comparable therewith, except that phosphorus and a little more silicon has been introduced into it.

Table 2: Compositions of samples of reference steel and of a steel according to the invention (in  $10^{-3}\%$ )

	C	Mn	Si	Cr	Mo	Ni	Cu	V	P	S	Al
reference	392	1383	523	193	29	87	118	88	8	56	25
invention	396	1405	620	158	21	85	151	89	96	85	2

Figure 1 represents the ratio of liquid phase to solid phase in these steels as a function of the temperature. For the reference steel the measured solidus temperature is  $1415^{\circ}\text{C}$  whilst it is  $1375^{\circ}\text{C}$  for the steel according to the invention. The measured liquidus temperatures are respectively  $1525$  and  $1520^{\circ}\text{C}$ . The addition of phosphorus and silicon has therefore had a significant effect on the solidus temperature only, but that has been sufficient to widen the solidification range substantially (by  $35^{\circ}\text{C}$ ). It should also be noted that the temperature range in which the liquid fraction of the steel is included between 10 and 40%, and which is usually considered the most favourable for thixoforging, is:

- for the reference steel, from  $1437$  to  $1468^{\circ}\text{C}$ ;
- for the steel according to the invention, from  $1427$  to  $1463^{\circ}\text{C}$ .

Therefore a lowering of this range of the order of  $5$  to  $10^{\circ}\text{C}$  and a widening of its extent by  $5^{\circ}\text{C}$  is observed, all things which lead in the direction of less stress on the tools during thixoforging and greater ease of obtaining conditions favourable to good progress of the operation. This effect would be enhanced if the quantity of phosphorus added were increased, or if other segregating elements were also added within the limits which have been stated.

Table 3 shows the composition of a reference steel and of a steel according to the invention which is comparable thereto, except that phosphorus, silicon, manganese (to compensate for the addition of silicon so as to maintain a suitable ratio Mn%/Si%) and sulphur have been introduced into it.

Table 3: Compositions of samples of a reference steel and of a steel according to the invention (in 10<sup>-3</sup>%)

	C	Mn	Si	Cr	Mo	Ni	Cu	P	S	Al
reference	0.377	0.825	0.19	0.167	0.039	0.113	0.143	0.007	0.009	0.022
invention	0.396	1.405	0.62	0.158	0.021	0.085	0.151	0.095	0.085	0.002

Figure 2 shows the ratio of liquid phase to solid phase in these steels as a function of the temperature. For the reference steel, the measured solidus temperature is 1430°C whilst it is 1378°C for the steel according to the invention. The measured liquidus temperatures are respectively 1528°C and 1521°C. The solidification range has therefore been widened by 45°C. The temperature range in which the solid fraction of the steel is included between 10 and 40% is:

- for the reference steel, from 1470 to 1494°C,
- for the steel according to the invention, from 1428 to 1464°C.

Therefore a lowering of this range of the order of 30 to 42°C and an increase in its extent by 12°C is observed.

With regard to the determination of the solidus and liquidus temperatures to be taken into account for carrying out the invention, it should be noted that they cannot always coincide with those which are calculated on the basis of the composition of the steel with the aid of formulae conventionally available in the literature. In fact, these formulae are valuable in the

case of passage from liquid steel to solid steel during solidification and cooling of the steel and for cooling rates of several degrees per minute.

In the case of measurements carried out with a view to application to thixoforging, the measurements must be carried out by starting from the solid steel and progressing towards the liquid steel, that is to say in the case of heating then of fusion of the steel. The tests are also carried out with conditions of increasing the temperature of the order of several tens of degrees per minute, corresponding to the conditions of heating prior to the thixoforging operation.

Conventionally, the thixoforging operation carried out on steels according to the invention must be preceded by heat treatment for globulisation of the primary structure of the billet if a globular structure is not already present and if experience shows that it cannot be obtained during heating of the billet with a view to thixoforming it. Obtaining such a globular structure before thixoforging for a steel of given composition and history may be verified if the billet is cooled suddenly before proceeding to thixoforging it. The structure is then observed as it was before the cooling.

With regard to the cooling of the part following thixoforging, this cooling must be carried out in still air and not in a forced manner in the case (frequent for this type of part) where the part has very substantial variations in cross-section, for example thin walls (1 to 2 mm) are connected to thick zones 5 to 10 mm or more). The use of blown air is prohibited in this case because then there is a risk of introducing very substantial residual stresses between thin walls and thick zones. This would result in surface defects degrading the properties of the thixoforged part.

In certain cases it may be necessary to slow down the cooling of the parts so as to favour the structural homogeneity of the different parts thereof. For this purpose the part can be passed into a tunnel regulated in temperature within the range 200-700°C for example.

However, if the thixoforged part does not exhibit such substantial variations in cross-section it may be tolerable to effect cooling in blown air. Such cooling may favour obtaining a homogeneous metallurgical structure in the cross-section of the part and good mechanical characteristics.